

Use of a silicone surfactant of the alkyl dimethicone copolyol type in the preparation of solid cosmetic water-in-oil emulsions and solid water-in-oil emulsions thus obtained.

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The present invention relates to the use of a silicone surfactant of the alkyl dimethicone copolyol type in the preparation of solid cosmetic water-in-oil emulsions and to solid cosmetic water-in-oil emulsions, in particular foundations.

Water-in-oil (W/O) emulsions are commonly used in the cosmetics field as they make it possible to form a film at the surface of the skin which prevents transepidermal water loss and protects the skin from external attacks.

In view of the requirements of consumers with regard to emulsions of this type, which must simultaneously have a good cosmetic quality, which is reflected in terms of appearance, of texture, of ease of application and of good protective properties, good hold and good resistance to sweat and to sebum, it is highly advantageous to be able to obtain emulsions exhibiting all these properties without exhibiting the disadvantages of conventional W/O emulsions.

25 Solid emulsions of the foundation type generally comprise fatty substances, such as solid

waxes and oils, water and a particulate phase generally composed of fillers and pigments.

However, these compositions, when they are applied to the skin, exhibit the disadvantage of transferring, that is to say of being at least partly deposited, while leaving a trace, on certain substrates with which they can be brought into contact, in particular an item of clothing or the skin. This results in mediocre persistence of the film on the skin, requiring the regular renewal of the application of the foundation composition.

Another disadvantage of the compositions of the prior art is poor dispersion of the pigments, resulting in an emulsion which is not homogeneous.

Patent Application JP-A-03261707 discloses solid cosmetic compositions of the water-in-oil emulsion type comprising silicone oils, solid waxes and water which also comprise spherical powders.

The emulsifiers used can be organopolysiloxanes modified by polyoxyalkylenes, such as dimethicone copolyols.

The solid emulsions obtained, namely foundations, are nonhomogeneous and coarse and have a microscopic appearance which does not conform and a macroscopic appearance which does not conform, and the pigments are not well dispersed.

The aim of the present invention is to overcome these disadvantages and the invention provides a solid homogeneous W/O emulsion, in which the pigments, dyes and oils are well dispersed, which is soft, which has good slip, which has very good hold and which has good persistence on the skin.

The Applicant Company has discovered, surprisingly and unexpectedly, that it is possible, by using a specific silicone surfactant of the alkyl dimethicone copolyol type, in combination with at least one oil and at least one wax, to obtain a solid water-in-oil emulsion which exhibits the desired characteristics and which also exhibits the advantage of not transferring.

The subject-matter of the invention is therefore the use of a silicone surfactant of the alkyl dimethicone copolyol type, the formula of which is shown hereinbelow, in the preparation of solid emulsions of the water-in-oil type comprising an aqueous phase emulsified by the said surfactant in a fatty phase comprising at least one oil and at least one wax.

Another subject-matter of the invention is a solid cosmetic water-in-oil emulsion, characterized in that it comprises an aqueous phase emulsified, using a silicone surfactant of the alkyl dimethicone copolyol

type with the formula shown hereinbelow, in a fatty phase comprising at least one oil and at least one wax.

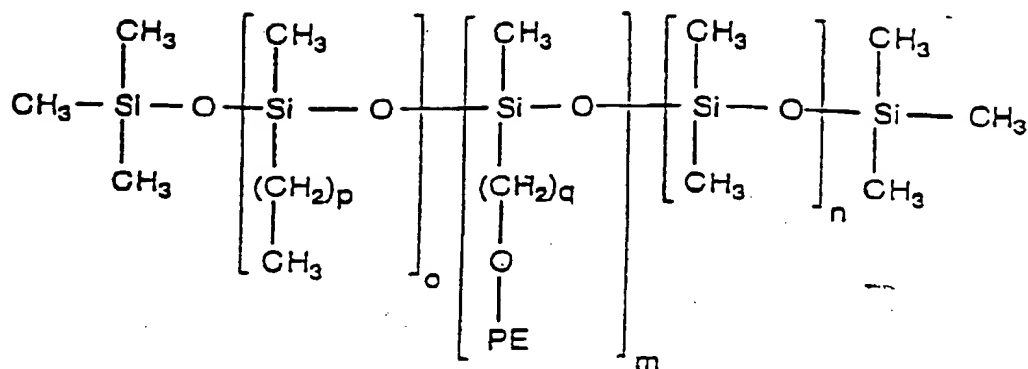
Another subject-matter of the invention is a process for making up the skin and/or scalp,
5 characterized in that a solid emulsion as defined above is applied to the skin and/or to the scalp.

The Applicant Company has found that the solid emulsion according to the invention is applied and is spread easily in a homogeneous way and exhibits
10 good moisturizing properties and good cosmetic properties since it is soft and has good slip. The film obtained also exhibits a light texture and remains comfortable to wear throughout the day.

Furthermore, the emulsion applied to the skin
15 exhibits the advantage of not migrating into the folds of the skin and/or the wrinkles of the face.

The emulsion according to the invention exhibits a homogeneous texture. Additives can be added to the emulsion according to the invention while
20 retaining a stable emulsion. The emulsion according to the invention is therefore compatible with a large number of cosmetic adjuvants.

The silicone surfactant of the alkyl dimethicone copolyol type used according to the
25 invention has the formula:



in which:

$$\text{PE} = (-\text{C}_2\text{H}_4\text{O})_x(-\text{C}_3\text{H}_6\text{O})_y-\text{H},$$

$$x = 0 \text{ to } 50,$$

$$y = 0 \text{ to } 30, \text{ } x \text{ and } y \text{ not simultaneously being } 0,$$

$$5 \quad o = 1 \text{ to } 100,$$

$$m = 1 \text{ to } 40,$$

$$n = 1 \text{ to } 200,$$

$$p = 1 \text{ to } 17 \text{ and}$$

$$q = 1 \text{ to } 5,$$

$$10 \quad \text{and preferably,}$$

$$o = 1 \text{ to } 25,$$

$$m = 1 \text{ to } 10,$$

$$n = 1 \text{ to } 100.$$

Such silicone surfactants are commercial products and mention may be made, as examples, of the compounds sold under the names:

- "Abil WE 09" by the Company Goldschmidt, which corresponds to the above formula and in which:

$$o = 21,$$

m = 4

n = 73,

- "Abil WS 08" and "Abil EM 90" by the Company
Goldschmidt,

5 - "218-1138" by the Company General Electric, which
corresponds to the above formula and in which

PE = $(C_2H_4O)_{12}-H$

o = 2

m = 8

10 n = 20

p = 9

q = 3.

Use is preferably made of the product "Abil
WE 09", which is a mixture of cetyl dimethicone
15 copolyol (CTFA name), of polyglyceryl-4 isostearate and
of hexyl laurate (33.3%/33.3%/33.4%).

The silicone surfactant, alkyl dimethicone
copolyol, is used in a proportion of 0.5 to 40% by
weight on the basis of the total weight of the emulsion
20 and preferably in a proportion of 2 to 12% by weight.

The fatty phase of the solid emulsion
according to the invention comprises at least one oil
and at least one wax.

The emulsion according to the invention
25 advantageously comprises 10 to 40% by weight and
preferably 18 to 30% by weight, with respect to the

total weight of the emulsion, of at least one oil.

The emulsion according to the invention preferably comprises at least one silicone oil.

Mention may be made, as examples of silicone
5 oils used in the invention, of:

- volatile cyclic silicones having from 3 to 8 silicon atoms and preferably 4 to 6, such as, for example, cyclotetradimethylsiloxane, cyclopentadimethylsiloxane or cyclohexadimethylsiloxane;
- 10 - cyclocopolymers of the dimethylsiloxane/methylalkylsiloxane type, such as "silicone FZ 3109" sold by the Company Union Carbide, which is a dimethylsiloxane/methyloctylsiloxane cyclocopolymer;
- 15 - volatile linear silicones having from 2 to 9 silicon atoms, for example hexamethyldisiloxane, hexylheptamethyltrisiloxane and octylheptamethyltrisiloxane;
- polyalkylsiloxanes with trimethylsilyl end groups,
20 preferably those for which the viscosity at 25°C is less than or equal to 0.06 m²/s, among which may be mentioned linear polydimethylsiloxanes, in particular those sold under the name "Dow Corning Fluid 200" by the Company Dow Corning, and alkylmethylpolysiloxanes,
25 such as cetyl dimethicone (CTFA name);
- phenylated silicone oils.

Volatile silicone oils are preferred for use in the invention.

Mention may also be made, as volatile oils preferably used according to the invention, of
5 hydrocarbon-comprising oils having from 8 to 16 carbon atoms and in particular volatile C_8 - C_{16} isoparaffin oils, such as isododecane, isodecane and isohexadecane.

The emulsion according to the invention can also comprise other oils and pasty fatty substances.

10 Pasty fatty compounds can be defined using at least one of the following physicochemical properties:
- a viscosity of 0.1 to 40 Pa.s, preferably 0.5 to 25 Pa.s, measured at 40°C with a Contraves TV rotary viscometer equipped with an MS-r3 or MS-r4 rotor at a
15 frequency of 60 Hz,

- a melting point of 25-70°C, preferably 25-55°C.

Mention may be made, among the other oils which can be used according to the invention, of:
20 - mineral oils, such as liquid paraffin or liquid petrolatum,
- animal oils, such as perhydrosqualene,
- vegetable oils, such as apricot oil, sesame oil, sweet almond oil, calophyllum oil, palm oil, castor
25 oil, avocado oil, jojoba oil, olive oil or cereal germ oil, such as wheat germ oil,

- branched C_8 - C_{16} esters, such as isohexyl neopentanoate,
- synthetic esters and ethers, such as oils of formula R_1COOR_2 in which R_1 represents the residue of a higher fatty acid comprising from 6 to 29 carbon atoms and R_2 represents a hydrocarbon-comprising chain comprising from 3 to 30 carbon atoms, such as purcellin oil, isopropyl myristate, 2-ethylhexyl palmitate, 2-octyldodecyl stearate, 2-octyldodecyl erucate, isostearyl isostearate, arachidyl propionate or 2-octyldodecyl benzoate; hydroxylated esters, such as isostearyl lactate, octyl hydroxystearate, octyldodecyl hydroxystearate, diisostearyl malate or triisocetyl citrate; or polyol esters, such as propylene glycol dioctanoate, neopentyl glycol diheptanoate, diethylene glycol diisononanoate and pentaerythritol esters;
- fatty alcohols having from 12 to 16 carbon atoms, such as octyldodecanol, 2-butyloctanol, 2-hexyldecanol, 2-undecylpentadecanol or oleyl alcohol;
- fluorinated oils, among which may be mentioned perfluoropolyethers, such as the products sold under the trade name "Fomblin" by the Company Montefluos, and fluorinated silicones, such as trifluoromethyl (C_1 - C_4)alkyl dimethicones, for example that sold under the trade name "X 22819" by the Company Shin Etsu; and
- their mixtures.

The cosmetic emulsion according to the invention advantageously also comprises 3 to 15% by weight and preferably 3 to 10% by weight, on the basis of the total weight of the emulsion, of at least one
5 vegetable, mineral, animal and/or synthetic wax.

Mention may be made, as waxes which can be used according to the invention, of waxes of animal origin, such as beeswax, spermaceti, lanolin wax and lanolin derivatives, vegetable waxes, such as carnauba,
10 candelilla, ouricury or japan wax, cocoa butter or cork fibre or sugar cane waxes, mineral waxes, for example paraffin wax, petrolatum wax, lignite wax or microcrystalline waxes or ozokerites, synthetic waxes, including polyethylene or polytetrafluoroethylene waxes
15 and waxes obtained by the Fischer-Tropsch synthesis, or alternatively silicone waxes, hydrogenated oils which are solid at 25°C, such as hydrogenated castor oil, hydrogenated jojoba oil, hydrogenated palm oil, hydrogenated tallow or hydrogenated coconut oil, and
20 fatty esters which are solid at 25°C, such as the C₂₀-C₄₀ alkyl stearate sold under the trade name "Kester Wax K82H" by the Company Koster Keunen.

The silicone waxes which can be used in the composition according to the invention can be
25 substituted linear polysiloxanes. Mention may be made, for example, of silicone polyether waxes or alkyl or

alkoxy dimethicones having from 16 to 45 carbon atoms. Mention may also be made of alkyl methicones, such as the C₃₀-C₄₅ alkyl methicone sold under the trade name "AMS C 30" by Dow Corning.

5 Use is preferably made of a wax or a mixture of waxes capable of conferring, on the solid emulsion according to the invention, a penetration force of greater than or equal to 50 grams (g).

 In the present application, this penetration
10 force is measured according to the following protocol: after preparation of the emulsion, the latter is cast in a dish and is maintained at 20°C for 24 hours. The penetration force is then measured on this solid emulsion using a Stevens texture-analysing device with
15 the TA24 measurement rotor, with a diameter of 4 mm, at a penetration rate of 0.5 mm/s and at a preselected penetration depth of 2 mm. The penetration force, expressed in grams, is read on the device.

 Use is preferably made of a wax chosen from
20 polyethylene wax, hydrogenated jojoba oil, ozokerite or their mixtures.

 More preferably, the emulsion according to the invention comprises polyethylene wax. More preferably still, the emulsion according to the
25 invention comprises a mixture of polyethylene wax and of hydrogenated jojoba oil.

The aqueous phase of the emulsion according to the invention can represent 0.5 to 60% by weight of the total weight of the emulsion.

It can comprise water or a floral water, such as cornflower water.

The aqueous phase according to the invention can comprise 0 to 14% by weight of lower C_2-C_6 monoalcohols and/or of polyols, such as glycerol, butylene glycol, isoprene glycol and propylene glycol, and agents for the stabilization of the emulsion, for example sodium chloride, magnesium dichloride and magnesium sulphate.

In addition, the emulsion according to the invention can comprise one or more thickening agents in concentrations preferably ranging from 0 to 6% by weight with respect to the total weight of the emulsion.

The emulsion according to the invention can also comprise a particulate phase which can comprise pigments and/or pearlescent agents and/or fillers commonly used in the field of cosmetics and make-up. A person skilled in the art will take care, however, to select these compounds for minimum transfer.

The pigments can be present in the emulsion in a proportion of 0 to 30% by weight with respect to the total weight of the emulsion and preferably in a

proportion of 2 to 20%. They can be white or coloured and inorganic and/or organic and have a conventional or nanometric size. Mention may be made, among inorganic pigments and nanopigments, of titanium, zirconium or cerium dioxides, as well as zinc, iron or chromium oxides, nano-sized titanium oxides and ferric blue. Mention may be made, among organic pigments, of carbon black, barium, strontium, calcium and aluminium lakes, and cochineal carmine.

The term "fillers" should be understood as meaning colourless or white, inorganic or synthetic, lamellar or nonlamellar particles. The term "pearlescent agents" should be understood as meaning iridescent particles, produced in particular by certain molluscs in their shells or else synthesized. These fillers and pearlescent agents are used in particular to modify the texture of the composition.

The fillers can be present in the emulsion in a proportion of 0 to 25% by weight with respect to the total weight of the emulsion, preferably 0 to 10%. Mention may in particular be made of talc, mica, silica, kaolin, Teflon, starch, boron nitride, Nylon powder (in particular Orgasol), polyethylene powder, copolymer microspheres, such as Expancel (Nobel Industrie) or Polytrap (Dow Corning), and silicone resin microbeads (Tospearl from Toshiba, for example).

The pearlescent agents can be present in the emulsion in a proportion of 0 to 20% by weight with respect to the total weight of the emulsion, preferably of 2 to 15%.

5 The pearlescent agents which can be used according to the invention are, for example, mica covered with titanium oxide, with iron oxide, with natural pigment or with bismuth oxychloride, as well as coloured titanium oxide-coated mica.

10 The emulsion according to the invention can additionally comprise any additive conventionally used in the cosmetics field, such as antioxidants, colorants, fragrances, essential oils, preservatives, cosmetic active principles, moisturizers, vitamins,
15 sphingolipids, sunscreen agents or fat-soluble polymers, in particular hydrocarbon-comprising polymers, such as polybutene, polyalkylenes, polyacrylates and silicone polymers compatible with fatty substances. Of course, a person skilled in the
20 art will take care to choose this or these optional additional compounds and/or their amount so that the advantageous properties of the composition according to the invention are not, or not substantially, detrimentally affected by the envisaged addition. These
25 additives can be present in the composition in a proportion of 0 to 15% by weight.

The emulsions according to the invention can be provided in the form of a cosmetic product and in particular in the form of a make-up product, in particular a foundation, a face powder, an eyeshadow or
5 a lipstick.

They can also be provided in the uncoloured form, optionally comprising cosmetic active principles.

The examples which follow serve to illustrate the invention without, however, exhibiting a limiting
10 nature. In these examples, the amounts are given as percentage by weight with respect to the total weight of the composition.

EXAMPLE 1:

15 The Applicant Company has prepared the following foundation:

PHASE O

20	- Hydrogenated jojoba oil	5.6%
	- Preservative	0.3%
	- Polyethylene wax (MW: 500)	2.9%
	- Polytetrafluoroethylene wax	7%

- Mixture of oxyethylenated and oxypropylenated poly(methylcetyl) (dimethyl) (methylsiloxane), polyglycerolated isostearate (4 mol) and hexyl laurate, sold under the name "Abil WE 09" by the Company Goldschmidt

9%

SILICONE S

- Cyclohexadimethylsiloxane (viscosity: $8 \times 10^{-6} \text{ m}^2/\text{s}$)

24.3%

PIGMENTS P

- Iron oxides
- Titanium oxide

2.9%

7.1%

PHASE W

- Sterilized demineralized water
- Propylene glycol
- Preservative
- Magnesium sulphate. $7\text{H}_2\text{O}$

38.5%

1%

0.4%

1%

The compounds of the phase O are weighed together and are heated to 100°C .

After homogenization, the phase is cooled to 80°C and

then the silicone S is added.

The pigments P are subsequently dispersed in the O+S mixture.

After homogenization, the phase W, preheated to 80°C,
5 is slowly added while stirring using a Moritz-type
stirrer and while retaining, during the addition, a
minimum temperature of 75°C.

The product is cast and a homogeneous compact
foundation is obtained which spreads well, which
10 results in a highly natural and very soft make-up,
which exhibits good cosmetic hold and which does not
transfer.

This composition has a penetration force,
measured on a Stevens device as described in the above
15 text, of greater than 50 grams.

**EXAMPLE 2: COMPARATIVE: Foundation according to the
prior art**

Example 5 of Application JP-A-03261707 was
20 repeated.

PHASE O

- Paraffin wax (mineral wax)

4%

	- Dimethicone copolyol, cyclopentasiloxane and water (10/88/2) mixture, sold under the name "Q2-3225 C" by the Company Dow Corning	30%
	- Camphor	0.1%
5	- Menthol	0.1%
	- Microcrystalline cellulose	3%

PIGMENTS P

10	- Titanium dioxide coated with polydimethylsiloxane	15%
	- Yellow iron oxide coated with polydimethylsiloxane	3%
	- Red iron oxide coated with polydimethylsiloxane	1%
15	- Black iron oxide coated with polydimethylsiloxane	0.2%

PHASE W

20	- Water	33%
	- Ethanol	5%
	- Polyethylene glycol	5%
25	- Methyl p-hydroxybenzoate	0.3%

A compact foundation is prepared according to
the above procedure.

A foundation is obtained which is heterogeneous in

colour, which is nonhomogeneous, which has pigments which are badly dispersed and which is rough and dry on application.

5 EXAMPLE 3:

The Applicant Company has prepared the following three compositions A, B and C, the nature of the wax being varied:

10 PHASE O

- Wax	6.3%
- Preservative	0.3%
- Mixture of oxyethylenated and	
oxypropylenated	
poly(methylcetyl) (dimethyl) (methyilsiloxane),	
polyglycerolated isostearate (4 mol) and hexyl	
laurate, sold under the name "Abil WE 09" by	
the Company Goldschmidt	9%

20

SILICONE S

- Cyclohexadimethylsiloxane (viscosity:	
$8 \times 10^{-6} \text{ m}^2/\text{s}$)	24.3%

25

PIGMENTS P

- Iron oxides 2.9%
- Titanium oxide 7.1%

5

PHASE W

- Sterilized demineralized water 38.5%
- Propylene glycol 1%
- 10 - Preservative 0.4%
- Magnesium sulphate.7H₂O 1%

with:

15	Composition	A	B	C
	Nature of the wax	Hydrogenated jojoba oil	Polyethylene	Carnauba
	Penetration force (in g)	74	101	11
20	Appearance after hot casting	Non-smooth solid	Solid	Soft
25	Observation under a microscope	Fine but not very even emulsion	Fine, even emulsion	Small crystals

These three emulsions were prepared as in Example 1. The penetration force was measured on a

Stevens device as described in the above text.

The emulsion C, which only comprises carnauba wax, does not make it possible to obtain a sufficiently solid composition.